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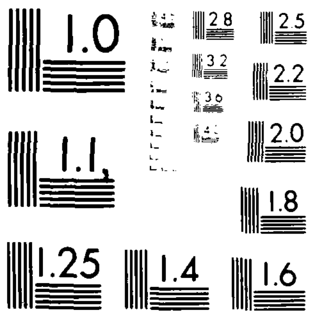

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COMPARATIVE TESTING OF POLYMER SOLUTIONS (U)

Part I: Preliminary Evaluation of a Rheometrics Fluids Rheometer

by

S.J. Armour and M.D. Gauthier

PCN No. 13E10

May 1983

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ABSTRACT

A comparison round of testing was organized to provide DRES with a low viscosity secondary standard for the measurement of viscosity and first normal stress difference in steady shear.

The current performance of the Rheometrics Fluids Rheometer, recently purchased by DRES, is compared with that of other Fluids Rheometers, with Weissenberg R-18 Rheogoniometers, with Instron Rotary Rheometers, and with concentric cylinder viscometers manufactured by Haake and Brookfield.

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ACKNOWLEDGEMENTS

The authors wish to thank their colleagues who participated in this first round of testing.

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COMPARATIVE TESTING OF POLYMER SOLUTIONS (U)

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INTRODUCTION

One of the requirements of the physical chemistry/rheology laboratory at DRES is to be able to accurately measure the rheological properties of solutions of interest to the Department of National Defence. These properties are viscosity (η) and first normal stress difference (N_1) in steady shear, dynamic viscosity (η') and shear storage modulus (G') in dynamic mode and elongational viscosity. Of these parameters, only viscosity in steady shear can be easily and accurately measured.

This series of comparison measurements was initiated primarily to supply the rheological laboratory with some solutions which could be used as secondary standards for the calibration of DRES instrumentation. Although several series of standards exist for the calibration of viscosity (see Appendix 1), only one standard is currently available for calibration of first normal stress difference. This is the 1490 Rheological Test Material which can be obtained from the National Bureau of Standards, Washington, D.C. (see Appendix 2). This material has the major disadvantage of having a high zero

shear viscosity (452 poise at 25°C) which places it outside of the range of many instruments. To the authors' knowledge, there are no standards for the calibration of dynamic viscosity and shear storage modulus.

Secondary benefits from this round of comparative testing include the opportunity of comparing the performance of the Rheometrics Fluids Rheometer, recently purchased by DRES, with other instruments currently in use in the rheological community.

SELECTION OF PARTICIPANTS

At the outset of this study, all the companies listed in the Analytical Chemistry Buyer's Guide as manufacturers of rheological test equipment were contacted and asked to test two samples. Four of the five major companies agreed to participate. The authors also asked colleagues who either had Rheometrics Fluids Rheometers or had expressed interest in comparison testing to participate. An additional series of measurements was made for DRES under contract (1). The participants listed in Table I have been designated 1 through 11 with DRES being participants 10 and 11.

TEST MATERIALS

In the initial round of testing two solutions were sent to each participant. The solutions were simply identified as DRES A and DRES B with no additional information as to their composition supplied.

DRES A was a solution of a copolymer of polymethylmethacrylate (41 g/L) in Dowanol DPM (dipropylene glycol methyl ether). The solution was prepared by placing the solvent in a clean linear polyethylene bottle, adding the polymer, and shaking vigorously to disperse the polymer. The solution was then rolled on a roller mixer for 72 hours at room temperature (2).

DRES B was the NBS 1490 Rheological Test Standard whose properties are given in Appendix 2.

TEST CRITERIA AND INSTRUCTIONS

Each participant was asked to adhere to the following test criteria:

1) All measurements were to be made at 25.0 ± 0.2°C.

2) Cone and plate configuration was to be used whenever possible. Data on cone angle, diameter and other relevant experimental parameters was to be specified.

3) Viscosity and first normal stress difference in steady shear were to be measured over as wide a shear rate ($\dot{\gamma}$) range as practical for the particular instrument used. The range $0.5\ s^{-1} \leqslant \dot{\gamma} \leqslant 250\ s^{-1}$ was to be covered for DRES A.

4) Dynamic viscosity and shear storage modulus in dynamic mode were to be measured over as wide a frequency (ω) range as possible. The range $0.5\ s^{-1} \leqslant \omega \leqslant 80\ s^{-1}$ was to be covered for DRES A.

5) The steady shear data was to be reduced according to the standard equations.

$$\dot{\gamma} = \frac{\Theta}{\beta} \qquad [1]$$

$$\tau_{12} = \frac{3T}{2\pi R^3} \qquad [2]$$

$$N_1 = \frac{2F}{\pi R^2} \qquad [3]$$

where $\dot{\gamma}$ = shear rate

β = cone angle

Θ = angular velocity

τ_{12} = shear stress

F = force required to keep the tip of the cone in contact with the circular plate

R = radius of cone

N_1 = first normal stress difference

T = torque

No corrections were to be made for inertial effects, secondary flow, etc. (see Reference 3 for a discussion of these equations).

6) All data was to be submitted in numerical form to facilitate easy comparison.

DATA SUBMITTED

The data submitted by the various participants is given, as submitted, in Appendix 3 for DRES A and in Appendix 4 for the NBS 1490 Rheological Test Material (DRES B). Unless otherwise stated, the data consisted of a single set of measurements which the authors assume to be representative of the general level of work of that participating laboratory. The steady shear data is displayed graphically in Figures 1 and 2 for DRES A and Figures 3 and 4 for the NBS 1490. The dynamic data for DRES A is displayed in Figures 5 and 6. No dynamic data was submitted for the NBS 1490.

DISCUSSION

The data submitted will be discussed in terms of instrument types. Specific comments will then be made comparing the performance of the Rheometrics Fluids Rheometer at DRES (RFR-D) with other Fluids Rheometers (RFR).

After consultations with several authorities in the field, the authors concluded that a properly functioning rheometer should be capable of reproducing viscosity data for calibration solutions to within ± 5% and first normal stress difference data for the NBS 1490 Rheological Test Material to within ± 15%.

In order to obtain accurate measurements of rheological parameters, it is essential that the temperature of the sample material be maintained at the desired temperature for the duration of the experiment. The temperature dependence of the viscosity of the NBS 1490 Rheological Test Material is given in Appendix 2. This data shows that a change of ± 1°C produces a 3.5% change in zero shear viscosity from the value obtained at T = 25°C. The same 1°C temperature change correspondingly produces a 7% change in first normal stress difference since this parameter is twice as sensitive to temperature change as viscosity. Consequently, only that data measured at 25 ± 0.5°C was included in the comparison.

When the authors included the NBS 1490 rheological test material as one of the two samples sent, they believed that most North American rheology laboratories routinely used this material to calibrate their instrumentation and, therefore, would

immediately recognize it once the viscosity-shear rate curve was run. This belief was based on comments by participant 5 who routinely uses NBS 1490 for calibration purposes. Consequently, participants 4 and 5 were not sent this material; a fact which subsequently precluded carrying out a full comparison of the performance of available instrumentation involved in the study.

For the NBS 1490 direct comparisons can be made between the data obtained in this series of measurements and that reported by the National Bureau of Standards (see Appendix 2). The viscosity data, with the exception of that given by the Haake instrument (participant 6), lie almost directly on the curve drawn through the NBS data.

For the first normal stress difference data, there is considerably more scatter. A % error = $[(N_1)_{NBS} - (N_1)_{measured}]/(N_1)_{NBS}$ has been calculated and is given in the final column of the tables in Appendix IV. Participant 1 who used an Instron Rotary Rheometer obtained excellent agreement with the NBS data with the maximum error being 6.2% and the average error being 2.8% for the eight measurements made. The second Instron instrument (participant 2) also exhibits good agreement with the NBS data at shear rates greater than $\dot{\gamma} = 2.1\ s^{-1}$ providing the authors' assumption that the participant has made an error of a factor of 10 in his calculations is valid.

The three RFRs (participants 8, 9, 11) give data which is lower than the NBS data by an average error of ~ 17% for particiants 8 and 9, ~ 21% for participant 11a. The agreement between these three instruments which all have the standard 100 g-cm torque transducers is acceptable. However participants 11b and c, using a 200 g-cm torque transducer gave errors of 33% and 39%, respectively.

The only Weissenberg R-18 (participant 3) also gave low data, exhibiting an error of ~ 37.5%. Consequently it would have been most informative if data had been obtained from the other two Weissenberg users.

In judging the performance of an instrument, the expertise and knowledge of the experimenter is also being judged. The authors, thus, do not know whether the apparent superior performance of the Instron Rotary Rheometer (participant 1) in measuring the NBS 1490 Rheological Test Material is due to its being a superior instrument or due to the expertise of the experimenter who is known by the authors to be very skilled. Consequently, before any valid comparison can be made between instrument types, it is advisable to have data from at least three different instruments of the same type in three different laboratories.

Since the values of the viscosity and first normal stress difference for DRES A are not known, only comments on the relative values can be made. In the case of viscosity, the scatter is considerably larger than that exhibited for the NBS 1490 test material. At $\dot{\gamma} = 10\ s^{-1}$ the range of viscosity measurements is between 8.0 and 9.4 poise while at $\dot{\gamma} = 100\ s^{-1}$ it is between 3.8 and 6.0 poise. The Instron data (participant 1) appears to lie in the center of the scatter with the three Weissenberg R-18s (participants 3, 4 and 5) lying above and one Fluids Rheometer (participant 9) lying below. The other Fluids Rheometer (participant 11) lies in the center of the scatter. The two concentric cylinder viscometers, the Haake (participant 6) and the Brookfield (participant 10) give data on the high and low side respectively.

The Brookfield viscometer which is a simple, inexpensive instrument was included in this comparison round because it has been widely used to measure the viscosity of toxic and/or corrosive materials. The fact that its performance is more than adequate is thus very reassuring.

For first normal stress difference the scatter in the data is again quite large; at $\dot{\gamma} = 100\ s^{-1}$ the range of N_1 measurements is between 2.7×10^3 and 5.3×10^3 dyne $\cdot$ cm^{-2}. The three Weissenbergs (participants 3, 4 and 5) give higher values than the two Fluids Rheometers (participants 9 and 11) with the Instron (participant 1) again in the center of the scatter.

Only three participants submitted dynamic data for DRES A (participants 3, 5 and 11c). The RFR-D data for both dynamic viscosity and shear storage modulus lies slightly above that of the two Weissenbergs (participants 3 and 5). However, the scatter in the measurements is quite small so that all three measurements can be considered to show good agreement.

On the basis of measurements made on DRES A, the RFR-D, equipped with the 200 g-cm torque transducer, 0.02 radian 5 cm diameter cone, appears to give acceptable viscosity and first normal stress difference data for $\dot{\gamma} \leq 160\ s^{-1}$. At $\dot{\gamma} > 160\ s^{-1}$, the values of N_1 appear to turn down. This downturn is not observed for the other RFR which also uses the same cone parameters, nor for any of the three Weissenbergs. The cause of the downturn, which is also observed with similar solutions when the RFR-D was equipped with the 100 g-cm torque transducer, is currently under investigation.

When the RFR-D is used to measure the NBS 1490 test material several additional questions about its performance are raised. For a given set of experiments (11a or 11c), the data for both viscosity and first normal stress difference exhibit acceptable

reproducibility. The viscosity data for all three sets of experiments (11a, 11b and 11c) is also quite acceptable when compared to the data given by the National Bureau of Standards. The first normal stress difference data, however, is always considerably lower than that given by the National Bureau of Standards. The best data, which is ~ 21% lower than expected, was obtained by participant 11a using the 100 g-cm torque transducer with a 0.04 radian 5 cm diameter cone. The data obtained with the 200 g-cm torque transducer is ~ 33% lower than expected for the 0.04 radian 5 cm diameter cone and ~ 39% lower for the 0.02 radian 5 cm diameter cone.

In his discussion of errors encountered in using cone and plate configuration for the measurement of first normal stress difference, Walters (4) states that inertial and secondary flow effects may be important. An estimate of inertial effects made using the formula:

$$\Phi = \frac{(3\varrho\Theta^2 R^2)}{10} \qquad [4]$$

where

Φ = inertial correction

ϱ = density of the solution

Θ = angular velocity

R = radius

shows that this contribution is insignificant for the NBS test material at $\dot{\gamma} = 31.5\ s^{-1}$. The secondary flow effects would probably be similar for the two 0.04 radian cones used by participants 11a and 11b and consequently, can not explain the differences observed. Reasonable agreement is also observed between the data obtained in experiments 11b and 11c where the cone angle is changed from 0.04 to 0.02 radians. Since the authors took great care in aligning the instrument, this common source of error can probably be ruled out. It thus appears that the 100 g-cm torque transducer is a significant improvement over the 200 g-cm torque transducer. The 100 g-cm transducer had originally been rejected because of problems with noise and drift. Further experiments are underway to verify that the transducer is indeed the cause of the discrepancy between these sets of measurements.

If the RFR-D can consistently reproduce the data for the NBS 1490 Rheological Test Material to within ± 5% for viscosity and ± 15% for first normal stress difference, it will be judged to be performing satisfactorily. It has already met the viscosity criteria

and for one transducer-cone combination is approaching the first normal stress difference criteria. The other two RFRs included in this comparison round are very close to being acceptable, having average errors for N_1 of $\sim 17\%$. Consequently, it is felt that the RFR-D can be made to perform as well as these instruments.

SUMMARY AND CONCLUSIONS

A series of comparison measurements of viscosity and first normal stress difference in steady shear has been made for two polymer solutions. This round of testing has provided DREES with a secondary standard for steady shear measurements.

Insufficient data was submitted to provide a secondary standard for the measurement of dynamic viscosity and shear storage modulus in dynamic mode.

The performance of the Rheometrics Fluids Rheometer, recently purchased by DREES, was compared to that of two other Fluids Rheometers, to three Weissenberg R 18 Rheogoniometers, to an Instron 3250 Rotary Rheometer, to two Haake Viscometers (RV12, RV100) and to a Brookfield Synchrolectric Viscometer. The RFR-D was found to accurately measure viscosity but to give low values for first normal stress difference for the NBS 1490 Rheological Test Material. The other two RFRs, although performing better than the RFR-D, were also observed to give low values for this parameter. Consequently, it appears that the RFR-D, will always give low values for first normal stress difference. It may, however, be possible to get the performance of the RFR-D within the acceptability criteria of ± 15% for measurement of first normal stress difference [illegible] NBS 1490 Rheological Test Material.

RECOMMENDATIONS

A second round of comparison testing should be undertaken using four test solutions covering a wider viscosity and first normal stress difference range. Particular emphasis should be placed on obtaining comparison measurements of dynamic viscosity and shear storage modulus in dynamic mode.

REFERENCES

1. Contract No. 75/82 Defence Research Establishment Suffield — Ecole Polytechnique, Montreal.

2. S.J. Armour and M.D. Gauthier, "Rheological Properties of Polymer Solutions — Part I. Solutions of K125/DPM (U)". Defence Research Establishment Suffield, Suffield Report No. 362. (1983), in preparation.

3. R.B. Bird, R.C. Armstrong and O. Hassager, "Dynamics of Polymeric Liquids — Volume 1 Fluid Mechanics", John Wiley and Sons (1977).

4. K. Walters, "Rheometry", Chapman and Hall (1975).

TABLE I

LIST OF PARTICIPANTS AND TEST EQUIPMENT USED

Participant Number	Instrument	Instrument Parameters
1	Instron 3250 Rotary Rheometer	cone diameter 4 cm
2	Instron 3250 Rotary Rheometer	cone angle 0.0209 radians diameter 6 cm
3	Weissenberg R-18 Rheogoniometer	cone angle 0.0172 radians diameter 5 cm
4	Weissenberg R-18 Rheogoniometer	cone angle 0.0087 radians diameter 5 cm
5	Weissenberg R-18 Rheogoniometer	cone angle 0.0175 radians diameter 5 cm
6	Haake RV-100 Rotoviscometer	CV 100 measuring system ZA 15 ZA 30 sensor systems
7	Haake RV-12 Rotoviscometer	PK-100 measuring system PKV 0.0175 radian cone M 500 measuring head
8	Rheometrics Fluids Rheometer	100 g-cm transducer cone angle 0.04 radians diameter 5 cm
9	Rheometrics Fluids Rheometer	100 g-cm torque transducer cone angle 0.0209 radians diameter 5 cm
10	Brookfield Syncrolectric Viscometer at DRES	concentric cylinder configuration $R_i/R_o = 0.88$
11a	Rheometrics Fluids Rheometer at DRES	100 g-cm torque transducer cone angle 0.04 radians diameter 5 cm
11b	Rheometrics Fluids Rheometer at DRES	200 g-cm torque transducer cone angle 0.04 radians diameter 5 cm
11c	Rheometrics Fluids Rheometer at DRES	200 g-cm torque transducer cone angle 0.02 radians diameter 5 cm

APPENDIX 1

VISCOSITY STANDARDS

A. Brookfield Engineering Laboratories Inc.
Stoughton, Ma.
02072, U.S.A.

Type	Approx. Viscosity at 25° C (cp)
5	4.9
10	9.4
50	50
100	96
500	490
1000	990
5000	5000
12,500	11,850
30,000	30,000
60,000	57,500
100,000	100,000

B. Cannon Instrument Company
P.O. Box 16
State College, Pa
16801, U.S.A.

Type	Approx. Viscosity at 25°C (cp)
S3	3.2
S6	7.7
S20	38
S60	109
S200	407
S600	1250
S2000	4550
S8000	24,200
S30,000	71,100

APPENDIX 1

VISCOSITY STANDARDS

C. Physikalisch-Technische Bundesanstald
Braunschweig,
West Germany

Type	Approx. Viscosity at 25° C (cp)
1B	0.9
2A	1.92
5B	4.00
10A	7.18
10B	10.6
10D^{+}	12.0
20C	17.1
20E	34.2
50C	66
100D^{+}	96
100C	114
200A	161
200G^{+}	240
500B	300
500F	450
500E^{+}	595
2000F	1290
2000E	2000
5000A	2900
10,000A	5800
10,000B	11,100
20,000D	19,900

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

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APPENDIX 2

National Bureau of Standards Certificate

Standard Reference Material 1490

Polyisobutylene Solution in Cetane (Viscosity and First Normal Stress Difference)

L. J. Zapas and J. C. Phillips

This Standard Reference Material is intended for the calibration and checking of instruments used in polymer technology and science for the determination of rheological properties of polymer melts or solutions. This particular solution was selected because of its relatively low flow activation energy and its long shelf-life stability.

Certified Values at 25.0 °C

Rate of Shear	Viscosity	First Normal Stress Difference
s^{-1}	Poise	N/m^2
0.0177	452	
0.0354	452	
0.0700	451	
0.12	447[a]	
0.28	432	
0.56	408	13.0[d]
1.11	356	33.5
2.22	308	77
4.45	241	160
8.80	179	310
17.7	125.5	540
35.5	83.0	910
70.0	53.5	1450
140.0	32.5[b]	
280.0	19.7[c]	

[a] This value was obtained using a calibrated capillary viscometer (Cannon Master) with the rate of shear taken at the capillary wall.

[b] Because there were indications of flow instability and viscous heating this value may be low.

[c] Though all precautions were taken to obtain this data point in less than 0.5 s, we cannot estimate the error.

[d] Due to the small value of the force, the scatter of the values was ±10%.

The technical and support aspects involved in the certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. K. Kirby.

Washington, D.C. 20234
December 21, 1977

J. Paul Cali, Chief
Office of Standard Reference Materials

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APPENDIX 2

The data on viscosity and first normal stress difference were obtained using a cone and plate arrangement. A cone of 1.5° was used for the viscosity measurements and a check with newtonian fluids under identical conditions (environmental & experimental) yielded values within less than ±1% of the determined value from our standard capillary viscometers, where the viscosity values are known to better than 0.5%. The first normal stress difference data are the average values of five runs obtained with different cones the angles of which were 1.5° and 2.29°. The extreme values, unless otherwise indicated, were within ±5 percent.

Values of the zero shear viscosity are given at other temperatures so that with the application of the time-temperature superposition the certified data can be extended within the range of these temperatures.

Temperature Dependency

Temperature	Zero shear viscosity	Density
°C	poise	g/mL
20.1	530	0.7819
25.0	452	0.7802
30.1	388	0.7784

APPENDIX 3

Data Submitted for DRES A

Part I - Steady Shear Data

Participant 1

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$)
5.00	9.43	43.8
7.89	9.11	91.5
12.49	8.33	179
15.61	8.02	250
19.73	7.68	385
24.98	7.35	
49.97	6.31	
78.89	5.59	
124.90	4.90	

Participant 2

- available load cell did not permit meaningful measurements to be made on this fluid.

Participant 3

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$)
4.62	8.57	76
46.20	6.90	1452
73.30	6.48	3311
116.00	5.70	6390
185.00	5.20	11,502

APPENDIX 3

Data Submitted for DRES A

Part I - Steady Shear Data

Participant 4

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$)
2.16	10.53	
3.41	10.11	
5.41	9.78	
8.57	9.36	
13.58	8.69	
21.56	8.14	299
34.12	7.36	789
54.12	6.71	1597
85.68	5.97	3293
135.84	5.35	5389
215.56	4.63	9181

Participant 5

- data from two separate determinations averaged by authors

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$)
0.27	12.11	
0.43	12.22	
0.68	11.63	
1.08	11.35	
1.72	10.93	
2.72	10.37	
4.31	10.16	
6.84	9.75	
0.84	8.95	
17.17	8.40	
27.22	7.55	439
43.14	6.80	968
68.37	6.23	2577
108.37	5.33	4374
171.75	4.75	7997
272.21	4.13	13,547

APPENDIX 3

Data Submitted for DRES A

Part I - Steady Shear Data

Participant 6

- data read off graphical output by authors

Shear Rate (s^{-1})	Viscosity (poise)
0.30	11.37
0.90	11.08
1.40	10.91
1.50	10.89
2.10	10.60
2.70	10.68
3.00	10.67
4.60	10.30
9.00	9.62
15.00	8.86
18.60	8.55
21.00	8.36
27.00	7.94
30.00	7.68
46.40	7.29
60.00	6.51
90.00	6.02
120.00	5.74
150.00	5.48

Participant 7

- was not given this sample to measure

Participant 8

- was not given this sample to measure

APPENDIX 3

Data Submitted for DRES A

Part I - Steady Shear Data

Participant 9

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$)
1.00	10.62	
1.59	10.02	
2.51	9.46	
3.98	9.00	
6.31	8.61	38
10.00	8.07	82
15.85	7.48	171
25.12	6.84	338
39.81	6.15	684
63.10	5.48	1460
100.00	4.77	2994
158.50	4.10	5250
251.20	3.47	8411

Participant 10

Shear Rate (s^{-1})	Viscosity (poise)
4.67	8.75
9.34	8.09
18.68	7.28
46.70	6.07
93.41	5.28

APPENDIX 3

Data Submitted for DRES A

Part I - Steady Shear Data

Participant 11c

Trial 1

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$)
2.70	9.94	
4.28	9.65	15
6.78	9.15	42
10.75	8.59	99
17.04	7.95	212
27.00	7.24	434
42.79	6.48	910
67.82	5.72	1855
107.50	5.01	3461
170.40	4.28	5499

Trial 2

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$)
10.00	8.56	104
15.85	7.96	199
25.12	7.27	381
39.81	6.56	807
63.10	5.84	1738
100.00	5.09	3195
158.50	4.35	5277
251.20	3.52	5964

APPENDIX 3

Data Submitted for DRES A

Part II - Dynamic Data

Participant 3

Angular Frequency (s^{-1})	Dynamic Viscosity (poise)	Storage Modulus ($dyne \cdot cm^{-2}$)
1.00	9.65	1.10
3.23	8.18	5.69
5.14	7.31	9.85
8.05	6.53	16.82
12.89	5.62	27.45
20.49	4.71	42.30
32.18	3.93	63.53
62.86	2.86	122.62

Participant 5

Angular Frequency (s^{-1})	Dynamic Viscosity (poise)	Storage Modulus ($dyne \cdot cm^{-2}$)
0.75	8.70	0.27
1.19	8.79	0.64
1.89	8.06	1.27
2.99	7.49	3.61
4.75	6.84	7.68
7.54	6.08	15.21
11.94	5.39	23.34
18.85	4.60	40.35
29.91	4.18	65.41
47.50	3.47	98.24

Participant 11c

Angular Frequency (s^{-1})	Dynamic Viscosity (poise)	Storage Modulus ($dyne \cdot cm^{-2}$)
2.70	8.79	5.23
4.28	8.07	9.77
6.78	7.23	17.23
10.75	6.29	28.51
17.04	5.34	44.78
27.00	4.45	67.47
42.79	3.64	97.50
67.82	2.94	136.40

APPENDIX 4

Data Submitted for DRES B

Participant 1

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$) measured	expected	% error
0.62	399.7	141	134	+5.6
0.99	371.4	275	288	-4.4
1.56	335.3	489	521	-6.2
2.50	293.9	866	893	-3.0
3.94	253.9	1432	1440	-0.6
7.89	192.4	2745	2789	-0.2
15.61	136.6	4853	4917	-1.3
24.98	104.5	7041	6947	+1.4

Participant 2

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$) measured	expected	% error
0.83	370.98	372	221	+68.3
1.32	334.40	677	423	+59.9
2.10	303.83	880	737	+19.4
3.32	266.25	1354	1209	+12.0
5.26	225.11	2031	1914	+ 6.0
8.34	186.56	2979	2930	+ 1.7
13.22	149.81	4197	4317	- 2.8
20.95	116.47	5077	6119	-17.0
33.20	88.40	8259	8606	- 4.0
52.63	66.53	11237	14132	- 2.0

- the authors assumed that the participant had made an error of a factor of 10 in his calculations and adjusted the data downward by this amount. The participant was not available to confirm this assumption.

APPENDIX 4

Data Submitted for DRES B

Participant 7

- read from graphical output by authors

Shear Rate (s^{-1})	Viscosity (poise)
8.80	170.8
17.70	122.9
35.50	86.2
70.00	55.7
140.00	31.6

Participant 8

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$)		
		measured	expected	% error
0.12	463.2			
0.19	454.9			
0.30	432.3			
0.48	415.3			
0.76	389.0	179	192	- 6.8
1.20	357.5	300	374	-19.8
1.90	319.5	531	658	-19.3
3.01	276.1	882	1091	-19.2
4.79	233.3	1432	1748	-18.1
7.57	191.4	2217	2687	-17.5

APPENDIX 4

Data Submitted for DRES B

Participant 3

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$) measured	expected	% error
4.62	217.0	1046	1688	-38.0
46.20	65.7	7087	11870	-40.3
73.30	50.4	9875	15000	-34.2

Participant 4

- did not measure this material

Participant 5

- did not measure this material

Participant 6

- read from graphical output by authors

Shear Rate (s^{-1})	Viscosity (poise)
0.30	500.0
0.56	460.7
0.90	432.9
1.11	410.8
2.22	345.9
4.45	276.4
8.80	208.0
12.00	181.3
17.70	148.3
21.60	132.6

APPENDIX 4

Data Submitted for DRES B

Participant 9

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$) measured	expected	% error
0.12	446.9			
0.21	441.0			
0.38	425.9			
0.67	397.5	156	154	+ 0.7
1.20	361.2	335	374	-10.4
2.13	315.6	606	749	-19.1
3.80	265.0	1083	1389	-22.0
6.75	211.8	1893	2421	-21.8
12.00	162.0	3064	3993	-23.3
21.34	118.7	5026	6202	-19.0

Participant 11a

Shear Rate (s^{-1})	Viscosity (poise) trial 1	trial 2	trial 3	trial 4	Mean	Standard Deviation
0.50	411.4	416.5	420.7	417.1	416.4	3.8
0.79	385.6	390.6	393.6	393.5	390.8	3.8
1.26	353.3	357.0	359.5	360.9	358.8	1.6
1.99	315.5	318.2	320.3	322.7	319.2	3.1
3.16	272.3	275.0	276.8	279.3	275.9	3.0
5.00	229.7	231.4	232.7	235.3	232.3	2.4
7.93	188.7	189.0	190.3	192.4	190.1	1.7
12.56	149.4	149.7	150.5	152.6	150.6	1.4
19.91	115.3	115.4	115.7	117.6	116.0	1.1
31.55	86.7	86.7	87.0	88.4	87.2	0.8

APPENDIX 4

Data Submitted for DRES B

Participant 11a

Shear Rate (s^{-1})	First Normal Stress Difference ($dyne \cdot cm^{-2}$) trial 1	trial 2	trial 3	trial 4	Mean	Standard Deviation	expected value	% error
0.50	78	83	67	60	72	10	86	-16.3
0.79	146	167	131	147	148	15	206	-28.2
1.26	268	305	268	295	284	19	397	-28.5
1.99	511	541	493	524	517	20	694	-25.5
3.16	858	912	847	881	875	29	1150	-23.9
5.00	1394	1488	1390	1445	1430	50	1820	-21.5
7.93	2244	2320	2196	2266	2260	50	2800	-19.4
12.56	3388	3492	3283	3432	3400	90	4140	-17.9
19.91	4980	5082	4812	5018	4970	120	5800	-14.3
31.55	7043	7133	6818	7083	7020	140	8260	-15.0

Participant 11b

Shear Rate (s^{-1})	Viscosity (poise)	First Normal Stress Difference ($dyne \cdot cm^{-2}$) measured	expected	% error
0.50	402.8	67	86	-22.1
0.79	375.0	132	206	-35.9
1.26	342.8	221	397	-44.3
1.99	306.9	417	694	-39.9
3.16	268.8	721	1150	-37.3
5.00	229.1	1201	1820	-34.0
7.93	188.8	1906	2800	-31.9
12.56	151.1	2874	4140	-30.6
19.91	117.7	4230	5800	-27.1
31.55	89.8	6098	8260	-26.2

APPENDIX 4

Data Submitted for DRES B

Participant 11c

Shear Rate (s^{-1})	Viscosity (poise) trial 1	trial 2	trial 3	trial 4	Mean	Standard Deviation
0.50	412.2	403.2	398.2	414.6	407	8
0.79	382.2	379.5	369.6	383.9	379	6
1.26	350.2	351.3	339.5	351.5	348	6
1.99	313.4	315.9	304.9	314.2	312	5
3.16	274.2	277.4	266.6	274.4	273	5
5.00	233.1	236.0	226.4	232.8	232	4
7.93	192.4	194.7	186.3	191.9	191	4
12.56	154.5	156.2	149.3	153.8	153.5	2.9
19.91	120.7	121.8	116.5	120.0	119.8	2.3
31.55	92.1	92.8	88.6	91.4	91.2	1.8

Shear Rate (s^{-1})	First Normal Stress Difference ($dyne \cdot cm^{-2}$) trial 1	trial 2	trial 3	trial 4	Mean	Standard Deviation	expected value	% error
0.50	57	67	41	37	51	14	86	-41.2
0.79	126	107	88	87	102	18	206	-50.5
1.26	232	225	180	176	203	29	397	-48.9
1.99	407	413	360	350	380	30	694	-44.8
3.16	697	736	654	637	680	50	1150	-40.8
5.00	1150	1231	1105	1096	1150	60	1820	-37.0
7.93	1818	1942	1763	1753	1820	90	2800	-35.0
12.56	2784	2951	2722	2706	2790	110	4140	-32.6
19.91	4107	4339	4030	4024	4130	150	5800	-28.9
31.55	5914	6226	5815	5827	5950	190	8260	-28.2

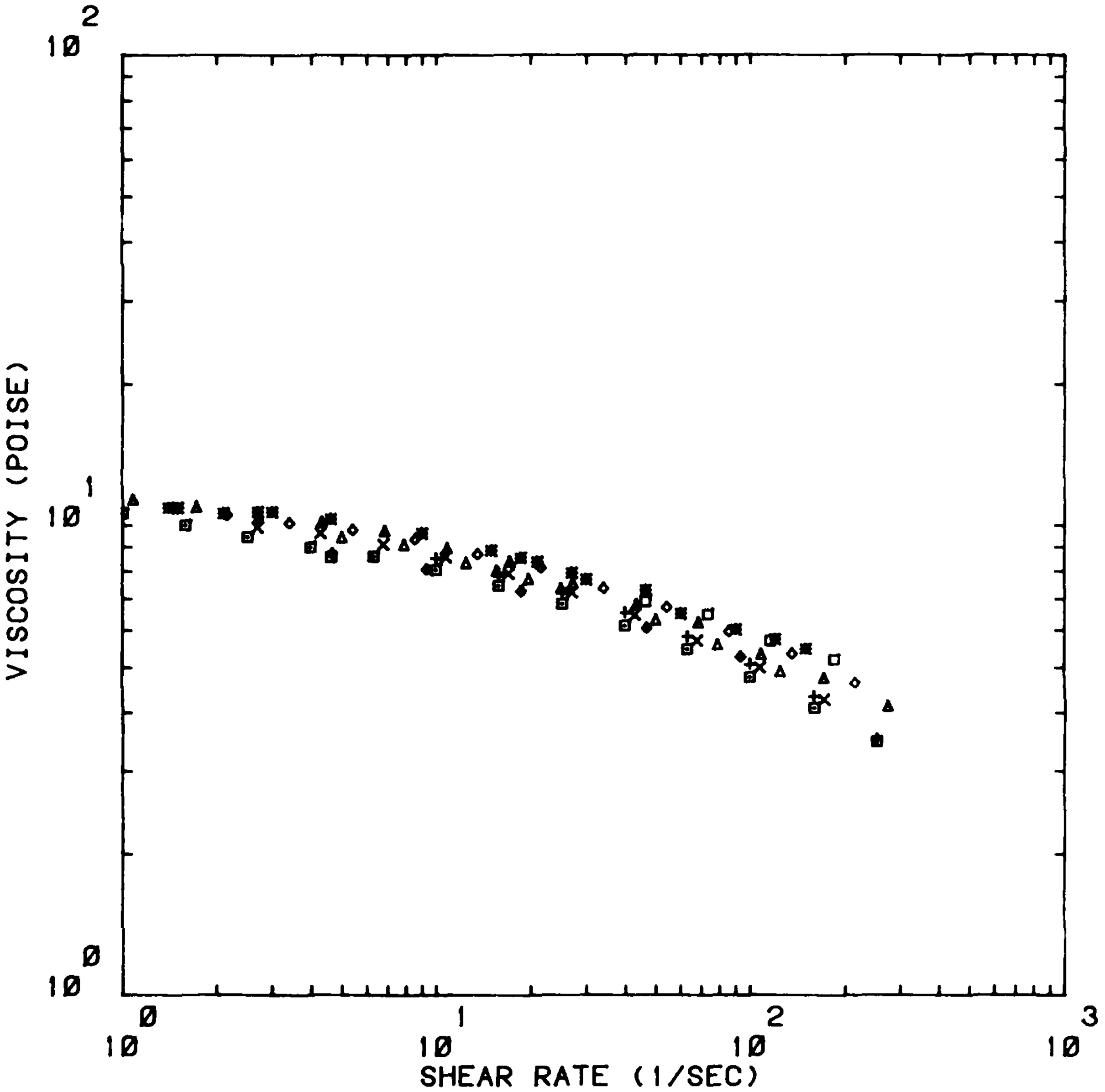

Figure 1. Viscosity Versus Shear Rate Data for DRES A.

△ Participant 1, □ Participant 3, ◇ Participant 4
▲ Participant 5, * Participant 6, ⊡ Participant 9
◈ Participant 10, × Participant 11c (trial 1),
+ Participant 11c (trial 2)

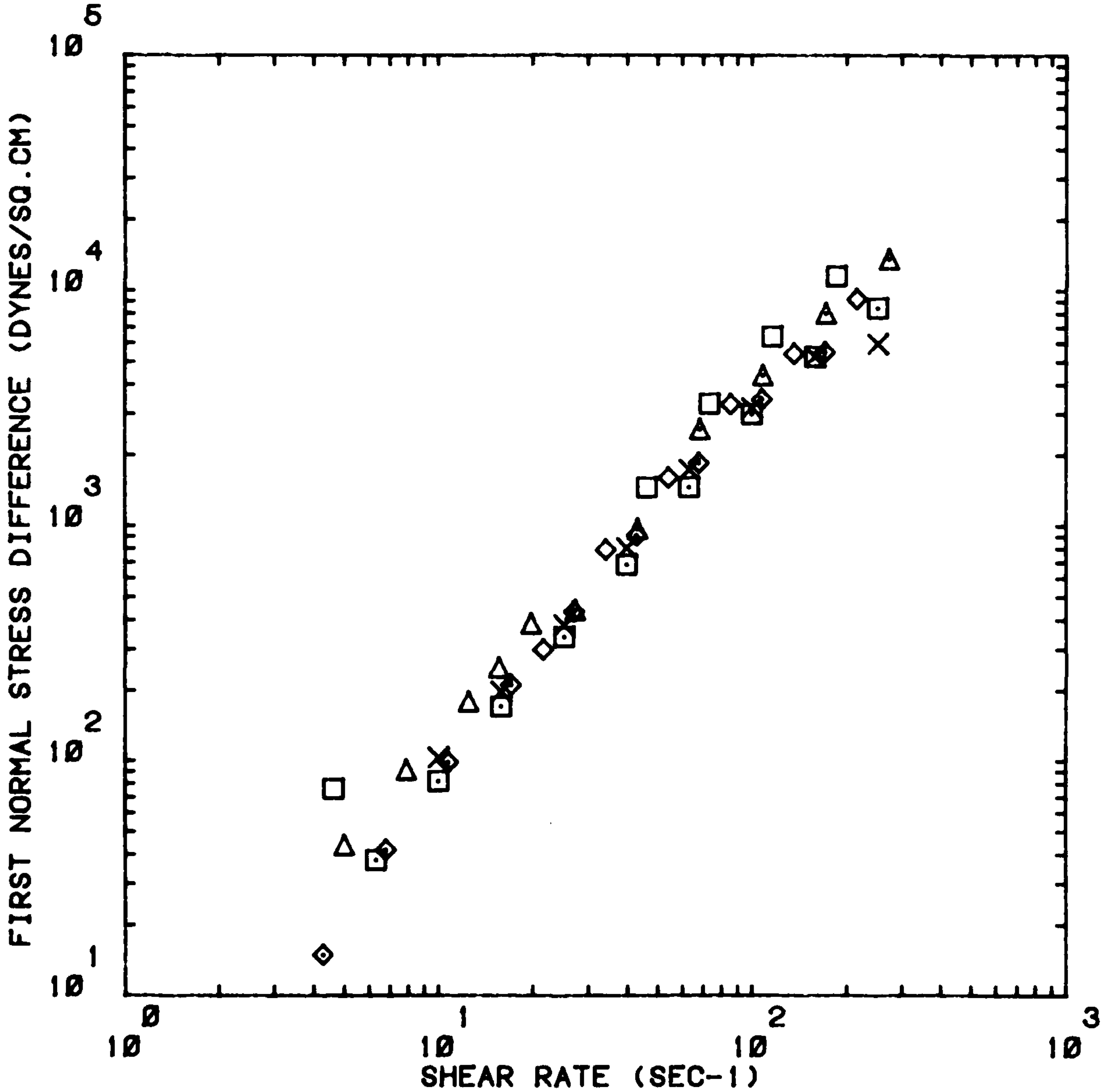

Figure 2. First Normal Stress Difference as a Function of Shear Rate for DRES A.

△ Participant 1, □ Participant 3, ◇ Participant 4
▲ Participant 5, ⊡ Participant 9,
◈ Participant 11c (trial 1), × Participant 11c (trial 2)

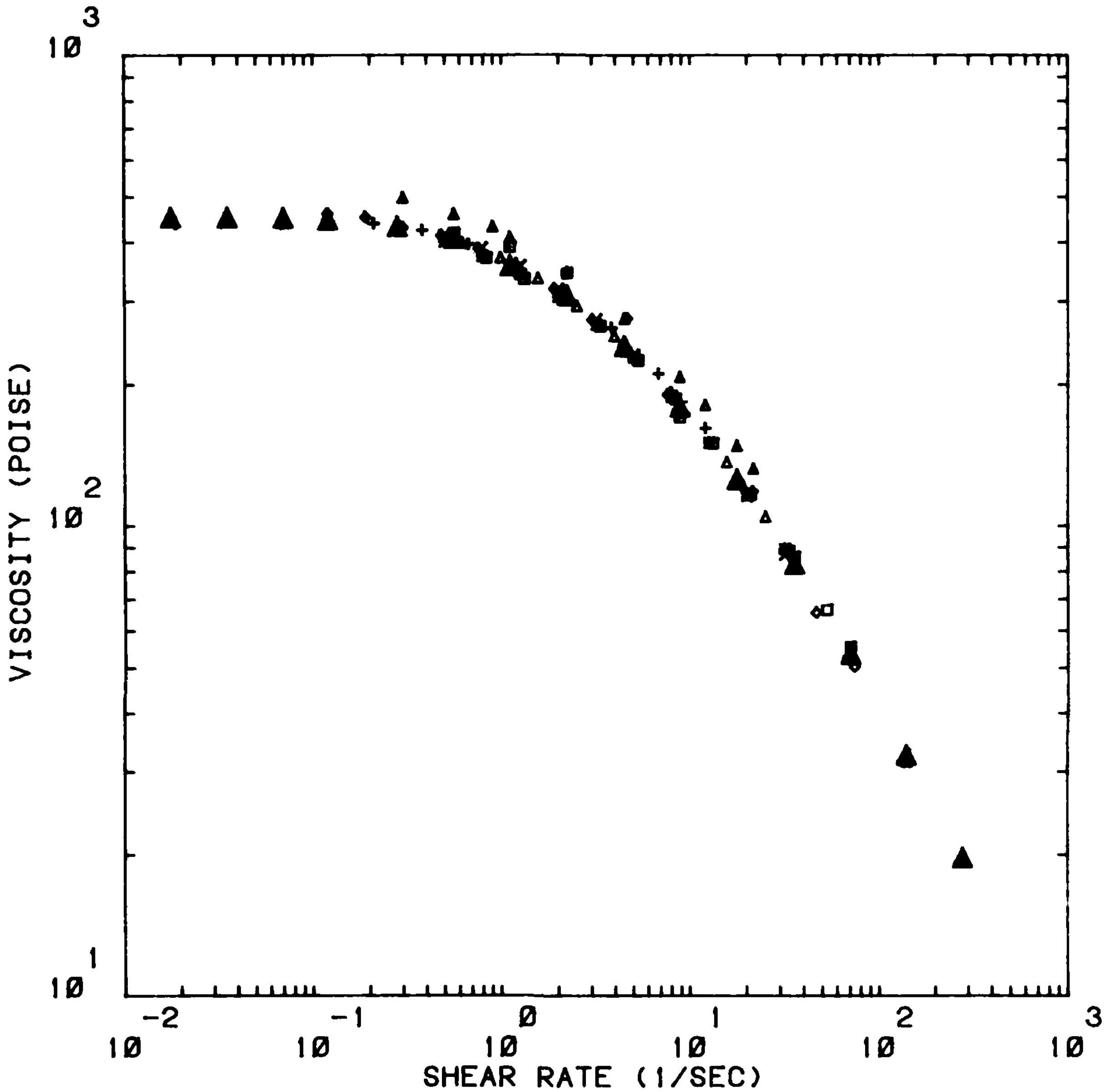

Figure 3. Viscosity Versus Shear Rate Data for NBS 1490

▲ NBS published data, △ Participant 1, □ Participant 2, ◇ Participant 3, ▲ Participant 6, ⊡ Participant 7, ◈ Participant 8, + Participant 9, × Participant 11a, * Participant 11b

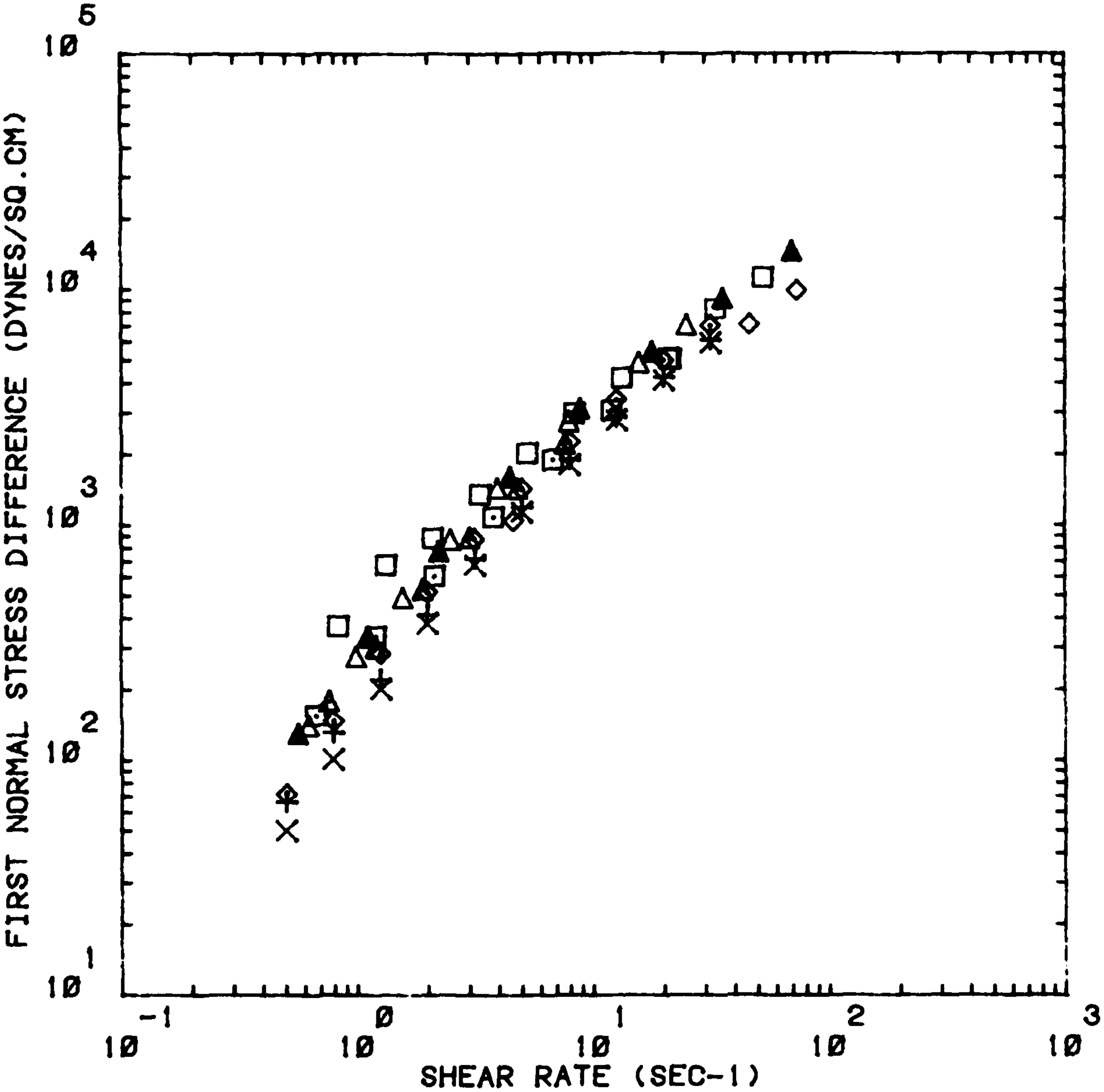

Figure 4. First Normal Stress Difference as a Function of Shear Rate for NBS 1490.

▲ NBS published data, △ Participant 1, □ Participant 2, ◇ Participant 3, ▲ Participant 8, ⊡ Participant 9, ◈ Participant 11a, + Participant 11b, × Participant 11c

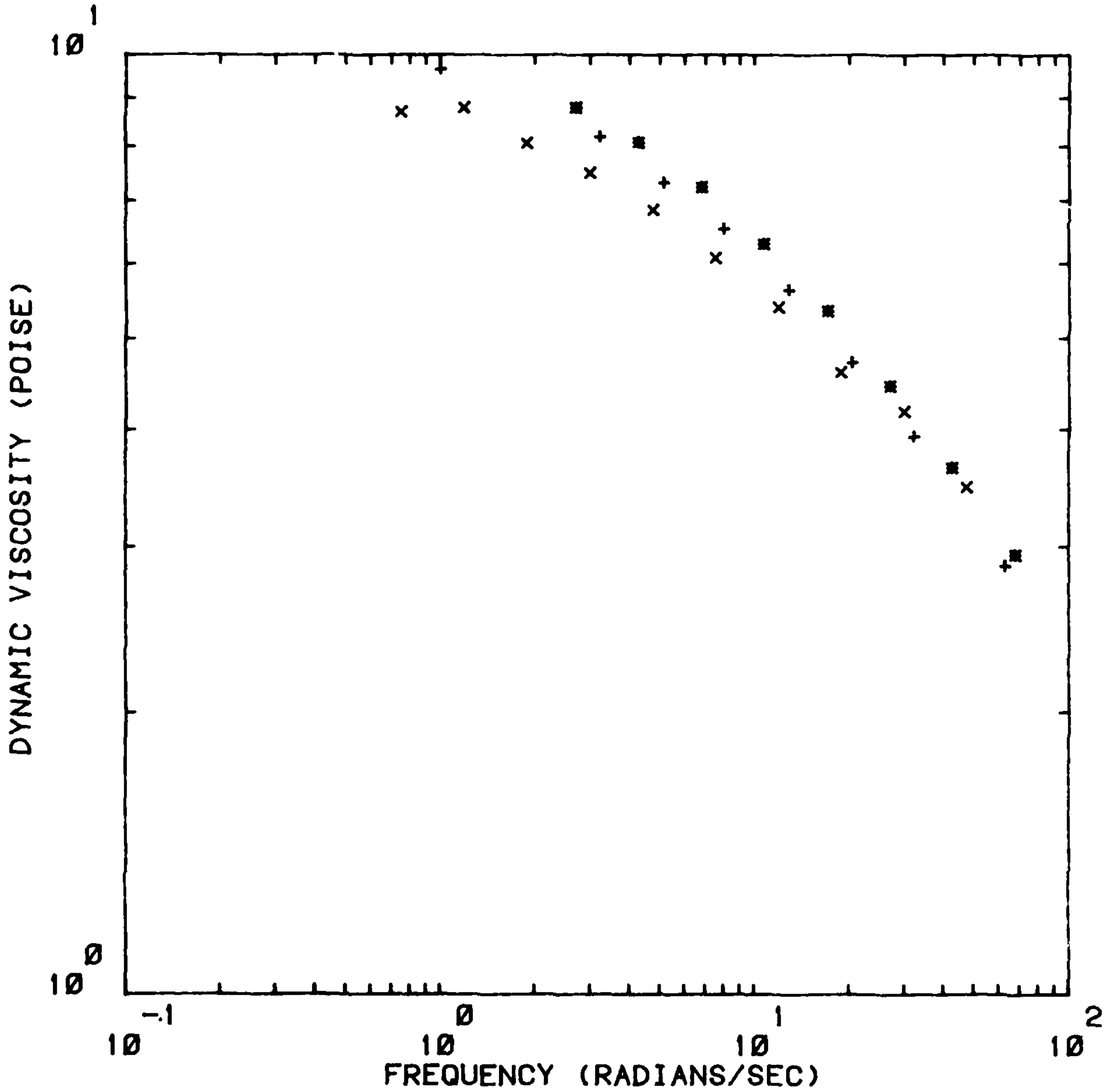

Figure 5. Dynamic Viscosity Versus Frequency for DRES A.
+ Participant 3, × Participant 5, * Participant 11c

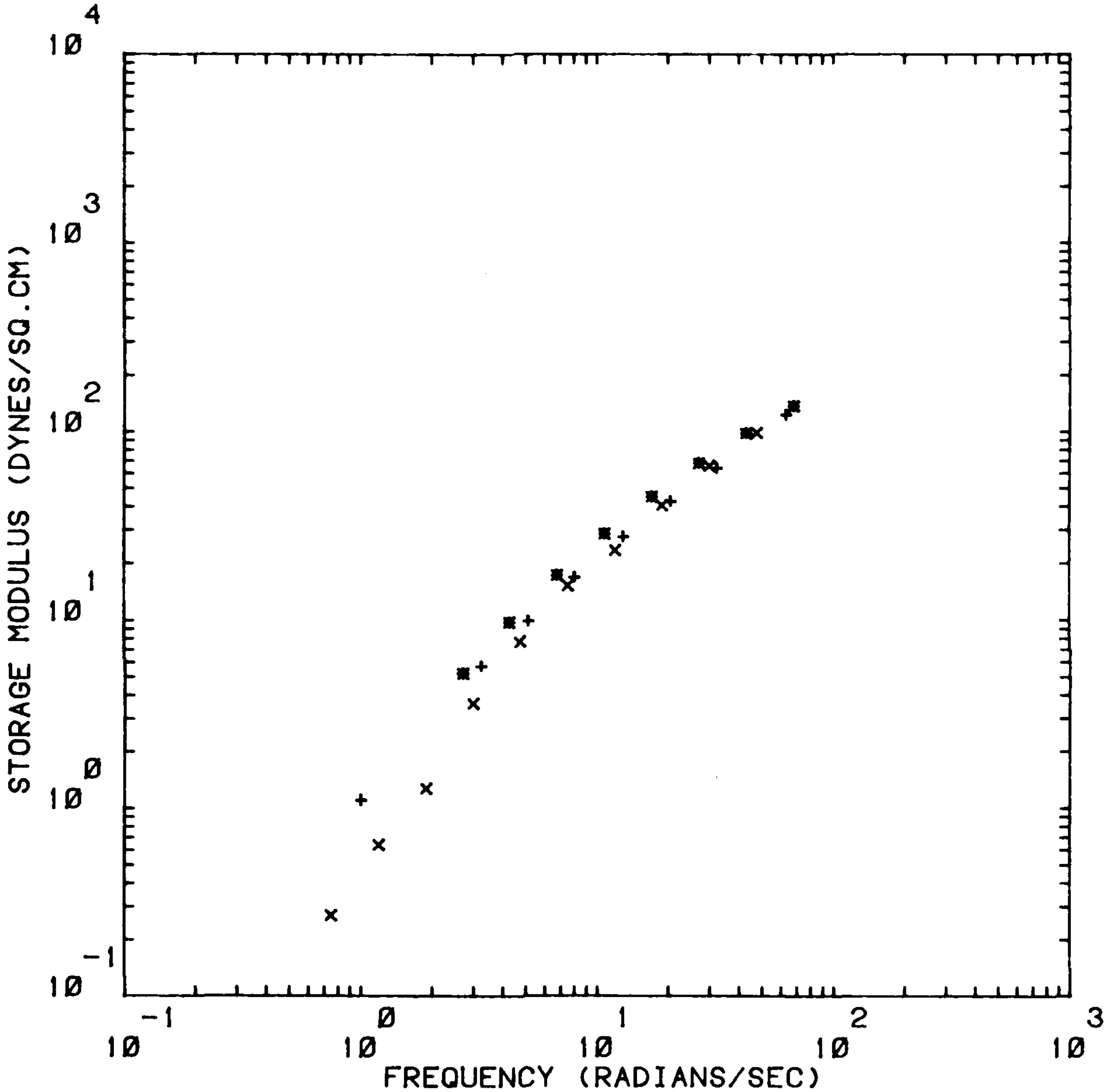

Figure 6. Storage Modulus as a Function of Frequency for DRES A.

+ Participant 3, × Participant 5, * Participant 11c

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13 ABSTRACT

A comparison round of testing was organized to provide DRES with a low viscosity secondary standard for the measurement of viscosity and first normal stress difference in steady shear.

The current performance of the Rheometrics Fluids Rheometer, recently purchased by DRES, is compared with that of other Fluids Rheometers, with Weissenberg R-18 Rheogoniometers, with Instron Rotary Rheometers, and with concentric cylinder viscometers manufactured by Haake and Brookfield. (U)

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KEY WORDS

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Viscometer

Comparison Testing

Viscosity

First Normal Stress Difference

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